

ADMINISTRATIVE INFORMATION

1. **Project Name:** Cavity-Enhanced Gas Analyzer for Process Control Applications
2. **Lead Organization:** Los Gatos Research
67 East Evelyn Avenue
Suite 3
Mountain View, CA 94041
3. **Principal Investigator:** Manish Gupta
650-965-7874 (phone), 650-965-7074 (fax),
m.gupta@lgrinc.com
4. **Project Partners:** Dow Chemical,
Linh Le, 979-238-7355 (phone)

Analytical Specialties Incorporated
Trevor Knittel, 281-488-0409 (phone)
5. **Date Project Initiated:** 06/27/03
6. **Expected Completion Date:** 06/26/05

PROJECT RATIONALE AND STRATEGY

7. **Project Objective:** (Please provide 1-2 sentences describing the objective of this project.)

This DOE SBIR effort focuses on developing an ultrasensitive gas analyzer to provide rapid quantification of trace acetylene contamination in ethylene gas flows. This instrument, which is based on our proprietary Off-Axis ICOS technology, will permit olefin manufacturers to incorporate real-time industrial process control.

8. **Technical Barrier(s) Being Addressed:** (Please provide 1-3 sentences describing the problem(s) and/or barrier(s) limiting industrial energy efficiency which this project is addressing.)

Currently, ethylene manufacturers rely on gas chromatography (GC) to detect acetylene contamination. However, although GC is sufficiently sensitive, it has proven to be too slow to provide real-time process control and error prevention, costing the petrochemical industry millions of dollars per year in acetylene upsets, contamination of stored product, and under optimized processes.

9. **Project Pathway:** (Please provide a one-paragraph summary of the approach, or pathway, being used to address the barrier(s). Emphasize the overall strategic approach for the project, not individual R&D tasks.)

This SBIR effort entails the development of a cavity-enhanced analyzer for industrial process control applications. In order to meet this goal, we have devised a project pathway that involves making a laboratory instrument and developing data analyses routines before producing and testing a final instrument package. The pathway is outlined below:

- Fabricate a laboratory prototype and measure key hydrocarbon constituents and mixtures
- Develop a data analysis routine to determine the acetylene concentration in the presence of other interfering absorbers (e.g. ethylene, etc...)
- Manufacture a robust, field-deployable analyzer
- Perform extensive laboratory testing at Dow Chemical Company and make any necessary adjustments
- Deploy the unit in the plant and perform long-term testing in conjunction with a GC
- Adapt the instrument to measure multiple contaminants in petrochemical gas flows
- Test the multiplexed unit in a variety of applications, including process control and endpoint validation

10. **Critical Technical Metrics:** (Please indicate how success or failure will be measured for this project by stating the baseline technical metric(s) and the metric(s) needed for realization of the project objectives.)

Baseline metrics (GC):

- Detect 0-10 ppm acetylene in ethylene gas flows with 0.1 ppm precision
- Requires 120 seconds to make a single measurement
- Cost of ownership is approximately \$350k over 15 years

Project metrics:

- Detect 0-10 ppm acetylene in ethylene gas flows with 0.1 ppm precision
- Measurement time of less than 5 seconds
- Cost of ownership of less than \$150k over 15 years

PROJECT PLANS AND PROGRESS

11. **Past Accomplishments:** (Please summarize the major accomplishments and **key** milestones achieved to date. Note: May not be applicable for projects initiated in FY04.)

Los Gatos Research has already made considerable progress along the project pathway. Specific accomplishments include:

- Redesigning the Off-Axis ICOS system to be more robust and compact. The new unit is completely integrated and field-serviceable to permit long-term deployment in a process environment. The mirrors can be periodically replaced without any need for realignment or adjustment, further increasing the instrument lifetime.
- Working with Analytical Specialties Incorporated (ASI) to integrate the analyzer head into a complete instrument with gas handling and computing subsystems.
- Collaborating with ASI to develop control, acquisition, and analysis software.
- Delivering the prototype to Dow Chemical Company and performing exhaustive laboratory testing. These tests have already led to a variety of improvements in system performance.

12. **Future Plans:** (Please summarize the **key** milestones and deliverables with dates for the life of this project. A comprehensive activities schedule is not required.)

- Complete lab testing at Dow Chemical Company.
- Deploy the analyzer into the plant and perform long-term field testing. A GC will be placed in tandem with our instrument to provide an independent acetylene concentration.

- Adapt the system to measure multiple ethylene contaminants both in the process stream and final product.
- Test the multiplexed instrument

13. **Project Changes:** (Please describe changes in scope, approach or schedule during the past year in response to any unforeseen problems/issues or successes.)

As we approach the first completed year of this SBIR Phase II, there have been no changes in either the scope or schedule of the proposed work. However, latter tasks may be altered during the upcoming year to better address the needs of our collaborating commercial partners.

14. **Commercialization Potential, Plans, and Activities:** Describe the end-use application and market potential for the project, and the plans, progress, and partners for commercial application/adoption, where appropriate; identify what the product of the project will be and how this product will be introduced/disseminated to industry.)

Currently, there are about 220 ethylene producing facilities worldwide. Each facility contains 1-2 measurement points, providing a potential market of 200-400 instruments. Due to the very high value of this measurement, Los Gatos Research expects to achieve nearly complete market penetration in this field. We intend to continue working closely with our commercial partner to manufacture, market, and sell the final instrument. Validation studies being performed at Dow Chemical Company will be used during instrument marketing to demonstrate sensitivity, time response, and longevity.

15. **Patents, Publications, Presentations:** (Please list number and reference, if applicable. If more than 10, please list only 10 most recent.)

Publications:

- D. S. Baer, J. B. Paul, M. Gupta, and A. O'Keefe, "Sensitive Measurements in the Near-Infrared Region Using Off-Axis Integrated Cavity Output Spectroscopy," *Appl. Phys. B* **75** (2002) 261.

Presentations:

- D.S. Baer, M. Gupta, A. O'Keefe and J. Paul, "Recent advances in off-axis integrated cavity output spectroscopy," invited paper SPIE Annual Meeting, Seattle, WA (July 2002).
- D.S. Baer, M. Gupta, A. O'Keefe and J. Paul, "Off-axis integrated cavity output spectroscopy for industrial process monitoring," Sixteenth International Forum Process Analytical Chemistry, San Diego (January 2002).
- D.S. Baer, M. Gupta, A. O'Keefe and J. Paul, "Off-axis integrated cavity output spectroscopy for chemical and environmental analysis," Laser Applications to Chemical and Environmental Analysis, Optical Society of America Spring Topical Meetings, Boulder, CO (February 2002).